

Ultratrace Analysis of Surface Impurities in Photovoltaic-Grade Polysilicon by ICP-MS

Quantification of 58 elements per GB/T24582-2023 using an Agilent 8900 ICP-QQQ



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Introduction

Green and sustainable energy reduces environmental harm, lowers carbon emissions, and supports long-term ecological balance.¹ As energy demand rises and climate impacts intensify, transitioning from fossil fuels to renewable sources, such as solar, wind, hydro, geothermal, and biomass, has become essential for sustainability and energy security.² Solar energy is widely used through technologies such as photovoltaic (PV) cells, which convert sunlight into electricity, and solar thermal systems, which produce heat. PV cells, which are typically made from crystalline silicon, are key to solar technology. The efficiency, cost, and durability of the cells largely depend on the materials used.^{3,4}

PV architectures are fundamentally built upon high-purity silicon ingots—a monocrystalline or polycrystalline mass of semiconductor-grade silicon, typically formed via the Czochralski or directional solidification process.⁵ These ingots are sliced into thin wafers that serve as the substrate for solar cell fabrication. In this process, doped regions and junctions are engineered to facilitate charge carrier separation and enable the conversion of incident solar radiation into electrical energy via the PV effect.⁶ Figure 1 depicts the manufacturing process for a PV panel.

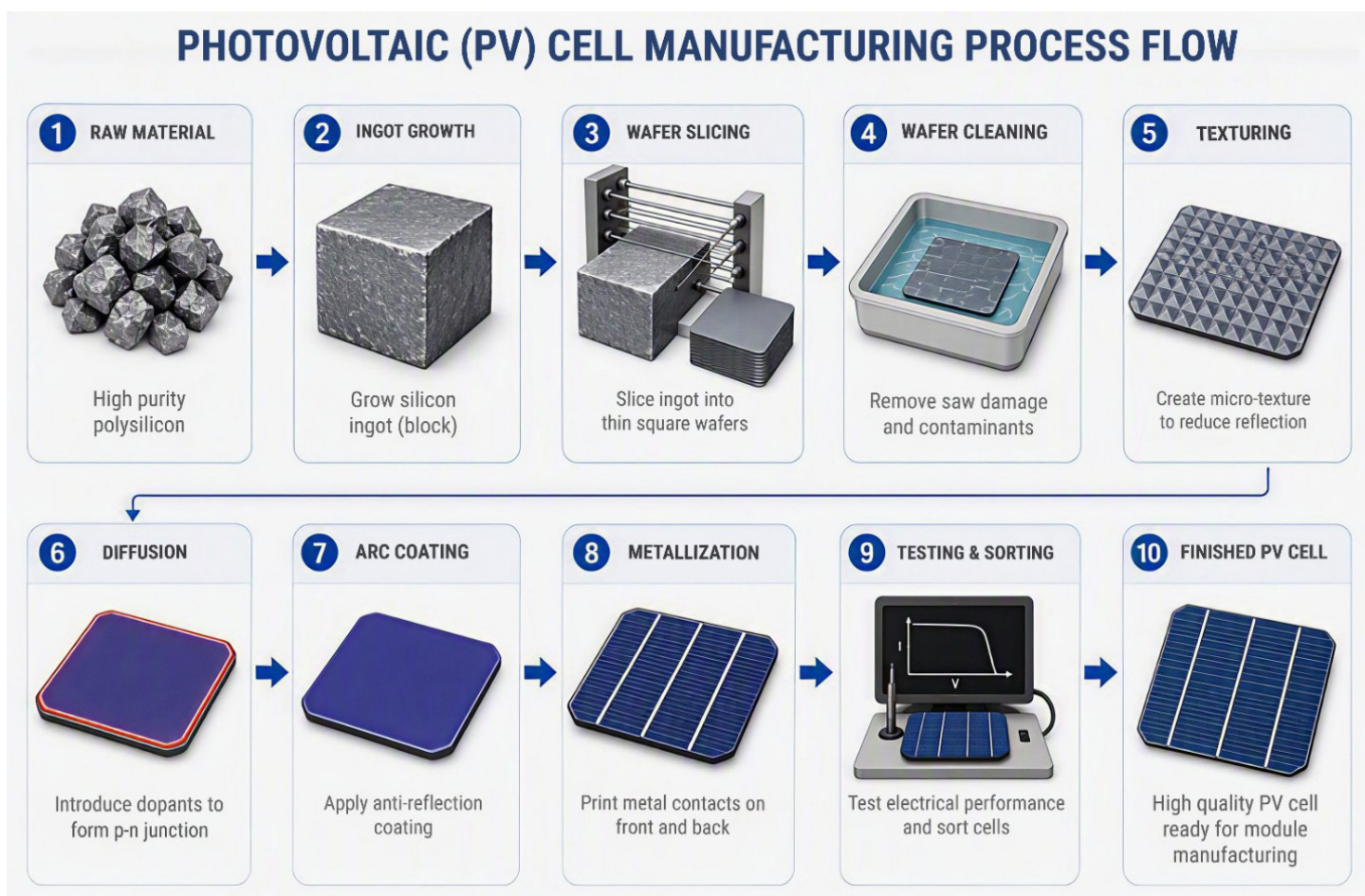


Figure 1. Photovoltaic panel manufacturing process.

Elemental impurity analysis of polysilicon materials is a critical quality control (QC) step in the production of high-efficiency PV cells. Silicon (Si), being the primary semiconductor material in most solar cells, must exhibit extremely high purity—typically 99.9999% (6N) or higher—to ensure optimal electronic properties. Even trace amounts of metallic impurities such as iron (Fe), copper (Cu), or nickel (Ni) can introduce deep-level defects in the silicon bandgap, acting as recombination centers that drastically reduce minority carrier lifetimes. This directly impacts the cell's ability to generate and collect charge carriers, leading to lower short-circuit current and reduced overall conversion efficiency. Precise elemental analysis also enables manufacturers to monitor and control contamination sources throughout the ingot growth and wafer fabrication processes.⁷

The Agilent 8900 Triple Quadrupole ICP-MS (ICP-QQQ) is an advanced analytical platform engineered for ultratrace elemental analysis in high-purity materials such as polysilicon used in PV manufacturing. Its unique tandem

mass spectrometry (MS/MS) configuration—comprising two quadrupoles (Q1 and Q2) separated by a collision/reaction cell—enables exceptional control over ion selection and reaction chemistry. Q1 acts as a mass filter to isolate target precursor ions, allowing only specific m/z values into the cell, while Q2 analyzes product ions formed after controlled reactions with gases such as O_2 , H_2 , or NH_3 . This design effectively eliminates complex spectral interferences, including polyatomic and isobaric overlaps that are particularly problematic in high-silicon matrices. As a result, the 8900 achieves sub-parts-per-trillion (ppt) detection limits for critical impurities like alkali and alkaline earth elements that are otherwise difficult to quantify accurately using conventional ICP-MS. Also, its high sensitivity, wide dynamic range, and robust plasma conditions ensure reliable quantification across varying concentration levels. These capabilities make the 8900 ICP-QQQ a useful instrument for QC and contamination monitoring in the semiconductor and solar industries, where even trace impurities can significantly impact PV cell efficiency and performance.

The instrument's cool plasma operation significantly reduces argon-based and matrix-derived interferences (such as ArO^+ and Si-based species), enabling accurate quantification of high first-ionization-potential elements in silicon matrices. Coupled with its exceptional matrix tolerance, the 8900 ICP-QQQ can directly handle highly concentrated, silicon-rich digests without signal suppression or plasma instability, maintaining analytical robustness and sensitivity. The system also supports fully automated analytical workflows, including application-specific method templates for semiconductor and PV-grade materials, ensuring high reproducibility, minimized operator intervention, and increased sample throughput. By delivering precise and reliable ultratrace impurity profiling, the 8900 ICP-QQQ plays a vital role in process monitoring, contamination control, and quality assurance for solar-grade silicon production. It thereby supports stringent industry specifications and enhancing PV device performance and yield.

Experimental

Reagents

All samples and standards were prepared in Ultrapur 60% nitric acid (HNO_3) and Ultrapur 48% hydrofluoric acid (HF) bought from Merck.

Sample preparation

Polycrystalline silicon samples were prepared for ultratrace metal impurity analysis using the acid extraction method described in the Chinese standard protocol, GB/T 24582-2023, *Test Method for Measuring Surface Metal Impurity Content of Polycrystalline Silicon – Acid Extraction-ICP-MS Method*. The process began with preconditioning, where all containers were cleaned using a 10% HNO_3 solution. Wash solutions were tested to confirm the absence of target elements, ensuring contamination-free conditions. During the sampling

stage, approximately 300 g of polysilicon—typically around 10 blocks—were selected. Samples were chosen for uniformity to ensure comparable surface areas and maintain repeatability, and triplicate sampling was performed to ensure statistical reliability. In the extraction process, 300 g of sample was weighed and placed into a clean container, followed by the addition of 250 mL of an extraction solution composed of HNO_3 , HF, and water in a 1:1:10 ratio.

The sample was fully submerged, and the extraction was carried out in a water bath maintained at 70 °C. After extraction, the post-extraction phase involved separating the supernatant solution from the solid residue. If necessary, centrifugation was applied at low temperature to aid in the separation. This method enabled accurate detection of ultratrace metal impurities in the polysilicon samples.

Calibration standards

Agilent multi-element calibration standards 1, 2A, 3, and 4 were used to prepare the calibration solutions for this study. A multi-element spike solution containing all elements was also prepared from the stock solutions.

Instrumentation

An Agilent 8900 Semiconductor configuration ICP-QQQ (#200 model) instrument was used for the analysis. It was equipped with an inert, HF-resistant sample introduction system comprising a MicroFlow PFA nebulizer, PFA spray chamber, endcap, connector tube, and a demountable torch fitted with a 2.5 mm internal diameter sapphire injector; although a platinum injector is recommended, it was not available. A platinum-tipped sampling cone was employed along with the s-lens, and a platinum-tipped, nickel-based skimmer cone designed for the s-lens.

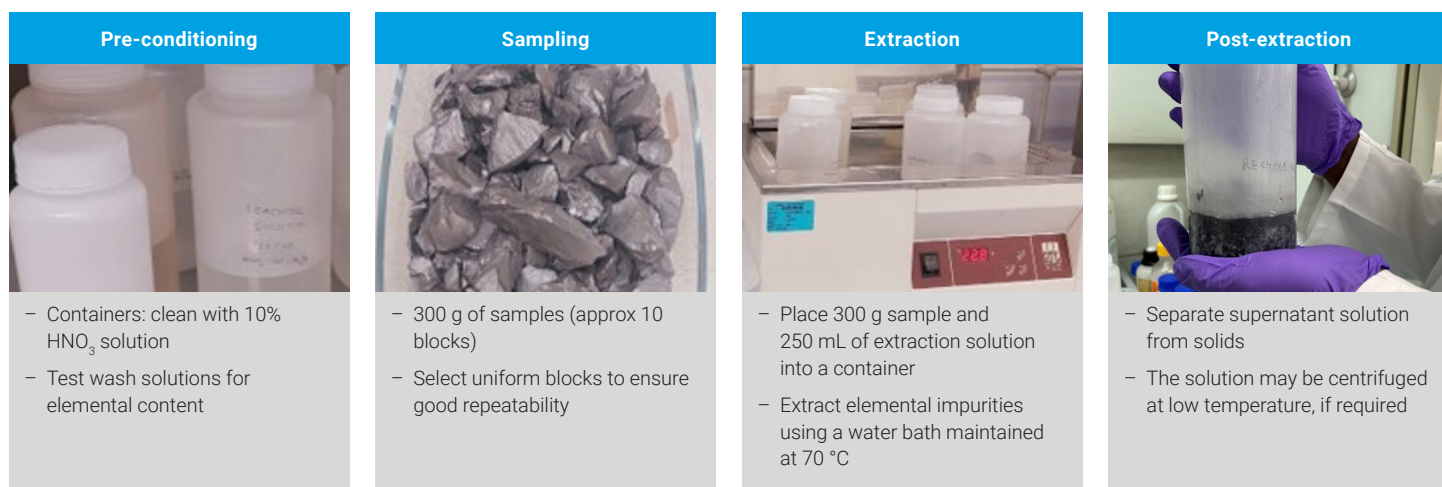


Figure 2. Sample preparation protocol for the extraction of elemental impurities from polycrystalline silicon as outlined in GB/T 24582-2023.

The PFA nebulizer and platinum-tipped sampling cone are standard components on the 8900 #200 model, designed for semiconductor applications. Additional sample introduction parts, including the PFA spray chamber, endcap, connector tube, and demountable torch, were provided as part of the Agilent Inert Kit. All listed sample introduction and interface components are also compatible with the Agilent 8900 ICP-QQQ #100 model, intended for advanced applications.

In high-silicon matrices, the formation of multiple polyatomic ions can lead to significant spectral interferences, particularly affecting key elements such as Fe, Ni, Cu, and Ge.⁸ To minimize background equivalent concentrations (BECs) and achieve accurate quantification, optimized ICP-QQQ acquisition conditions were applied for each analyte.

Using Agilent OpenLab ICP-MS instrument control software, a single multi-tune method enabled the measurement of different analytes under various cell gas modes. During data acquisition, the ORS⁴ reaction cell automatically switched gases and measurement modes, allowing fast and automated analysis using the most suitable conditions for each element. In this study, several reaction cell gases—including He, H₂, O₂, and NH₃—were employed as appropriate for the wide range of analytes analyzed.

Details of the configuration and principles of ICP-QQQ and MS/MS are explained elsewhere.⁹ The operating parameters of the 8900 ICP-QQQ are given in Table 1.

Table 1. Agilent 8900 ICP-QQQ operating conditions.

	Cool	Cool NH ₃	Cool H ₂	No Gas	He	HEHe	O ₂
Scan Type	Single	MS/MS		Single			MS/MS
RF Power, W	600		700	1500			
Sampling Depth (mm)	18			7.8			
Nebulizer Gas Flow Rate (L/min)	0.7						
Nebulizer Pump (rps)	0.1						
Spray Chamber Temp (°C)	2						
Make-up Gas (L/min)	0.75			0.56			
Extract 1 (V)	-150			4	4.8	3	4
Extract 2 (V)	-15			-230	-130		-230
Omega Bias (V)	-70			-150	-70		-150
Omega Lens (V)	2			10	5	4	10
Q1 Entrance (V)	-10			-5	-50		
Q1 Exit (V)	-5		-7	0	1	0	0
Deflect (V)	12	-3	-6	13	-3.2	0	6
Plate Bias (V)	-50		-100	-50	-60	-60	-60
He Cell Gas Flow Rate (mL/min)		1			5	9	
H ₂ Cell Gas Flow Rate (mL/min)			5				
NH ₃ Cell Gas Flow Rate (%)		20					
O ₂ Cell Gas Flow Rate (%)							30
OctP Bias (V)	-20	-10	-20	-8	-18	-18	-3
Axial Acceleration (V)	0	1.5	1	0	1	1	1.5
Energy Discrimination (V)	15	-5	0	5	3	5	-7

Results and discussion

Two silicon ingot samples, designated as Source A and Source B, were quantitatively analyzed using an external calibration strategy, allowing accurate determination of elemental concentrations across the targeted analyte range. For each element, BECs were calculated automatically using the OpenLab ICP-MS software. The BECs provide a direct assessment of background signal contributions under the selected MS/MS operating conditions. The data presented in Table 2 represent the mean of six replicate measurements for each sample, ensuring statistical reliability, with the corresponding BECs reported to evaluate background levels and interference control. The 8900 was operated in a standard laboratory environment, so lower BECs may be achievable under cleanroom conditions.

The generally low BEC values observed for most analytes highlight the high selectivity and effectiveness of the 8900

ICP-QQQ in MS/MS mode. This performance is especially significant for elements such as Fe, Ni, Cu, and Ge, which are prone to severe spectral interferences arising from silicon-based polyatomic ions in high matrix samples. The use of tailored reaction chemistries enabled efficient suppression or removal of these interferences, resulting in reduced background levels and improved signal-to-noise ratios.

Relatively higher BECs observed for a limited number of elements are likely attributable to external contamination sources, such as reagents and acids used during sample preparation or handling-related environmental exposure, rather than instrumental limitations. These observations underscore the importance of reagent purity and clean-handling protocols in ultratrace analysis. Overall, the results demonstrate the robustness of the 8900 ICP-QQQ approach for impurity profiling in silicon-rich matrices and confirm its suitability for stringent QC requirements in PV-grade material analysis.

Table 2. BECs and mean quantitative analysis results for polysilicon ingot samples from Sources A and B, $n=6$.

Element	Q1	Q2	Cell Gas Mode	BEC (ng/L)	Source A (ng/L)	Source B (ng/L)
Li		7	Cool	0.01	11.2	5.8
B		11	No Gas	1.35	5.7	86.8
Na		23	Cool	1.41	2982.6	3167.4
Mg		24	Cool	7.84	2083.3	3148.3
Al		27	Cool	0.46	<DL	<DL
K	39	39	Cool NH ₃	6.30	438.1	750.1
Ca	40	40	Cool H ₂	5.31	2175.2	1757.1
V	51	67	O ₂	1.00	7.4	10.4
Cr	52	52	Cool NH ₃	3.14	67.1	348.5
Mn	55	55	Cool H ₂	0.41	764.6	51.2
Fe	56	56	Cool H ₂	3.95	1434.3	2815.7
Co	59	59	O ₂	1.46	66.6	207.7
Ni		60	He	0.87	65.7	165.7
Cu	63	63	Cool NH ₃	3.31	150.6	0.0
Zn		66	He	5.34	661.5	742.7
Ga		71	Cool	0.73	45.0	58.1
Ge		72	He	3.05	<DL	<DL
As	75	91	O ₂	2.16	<DL	<DL
Se	78	94	O ₂	5.71	0.5	0.3
Rb		85	Cool	0.64	7.1	8.5
Sr	88	88	HEHe	0.53	46.1	46.8
Y		89	He	0.22	<DL	1.6
Zr	90	106	O ₂	0.22	6.3	175.1
Nb		93	He	0.27	<DL	<DL
Mo		95	He	1.09	<DL	<DL
Ru		101	He	0.84	<DL	<DL
Rh		103	He	2.31	<DL	<DL
Pd		105	He	1.44	<DL	<DL
Ag		107	He	2.72	<DL	<DL

Element	Q1	Q2	Cell Gas Mode	BEC (ng/L)	Source A (ng/L)	Source B (ng/L)
Cd		111	He	1.46	1.9	1.1
Sn		118	He	2.53	4.7	5.6
Sb		121	He	7.05	<DL	3.9
Cs		133	Cool	0.21	1.4	<DL
Ba	137	137	HEHe	0.50	1719.9	<DL
La		139	He	0.29	<DL	1.6
Ce		140	He	0.13	1.6	3.3
Pr		141	He	0.02	<DL	<DL
Nd		146	He	0.08	<DL	1.6
Sm		147	He	0.02	<DL	<DL
Eu		153	He	0.01	<DL	<DL
Gd		157	He	0.02	1.0	1.0
Tb		159	He	0.05	<DL	<DL
Dy		163	He	0.02	<DL	<DL
Er		166	He	0.01	<DL	<DL
Tm		169	He	0.00	<DL	<DL
Yb		172	He	0.00	<DL	<DL
Lu		175	He	1.33	1.0	0.8
Hf		178	He	0.68	<DL	<DL
Ta		181	He	0.11	<DL	<DL
W		182	He	0.43	153.0	316.4
Re		185	He	0.21	0.5	0.8
Ir		193	He	1.00	<DL	<DL
Pt		195	He	0.49	<DL	<DL
Au		197	He	4.62	<DL	5.3
Tl		205	He	1.30	0.5	0.8
Pb		208	He	7.20	21.0	18.1
Th		232	He	0.03	<DL	0.3
U		238	He	0.35	1.6	1.1

Spike-recovery experiments

The stability, robustness, and matrix tolerance of the analytical method were systematically evaluated through spike-recovery experiments, in which 0.1 µg/L of each target element was added to the silicon sample solutions. Recovery was assessed by comparing measured concentrations in the spiked samples against expected values, providing a direct measure of method accuracy and resistance to matrix-induced signal suppression or enhancement.

Spike-recovery performance for all analytes is shown as a bar chart (Figure 3). All recoveries were within the 80 to 120% acceptance limits, demonstrating the effectiveness of the ICP-QQQ operating conditions, including optimized MS/MS reaction chemistry and the use of the s-lens for enhanced ion transmission. These results indicate robust performance and reliable control of spectral interferences and matrix effects, enabling the accurate quantification of trace elements in high-silicon matrices.

Internal standard stability

Signal stability was systematically assessed by monitoring instrumental drift through the continuous measurement of selected internal standard elements, namely Y, Os, Lu, and In, as shown in Figure 4. These elements were chosen because they span a wide mass range and ionization characteristics,

making them suitable for detecting fluctuations across the analytical mass spectrum.

During a continuous analytical sequence extending over three hours, the instrument response for each monitored internal standard was recorded at regular intervals.

The normalized signal intensities for all four elements consistently remained within the predefined control limits of 80–120% relative to their initial responses. This range is widely accepted in quantitative ICP-based analyses as an indicator of stable instrument performance.

The absence of significant deviation beyond these limits indicates minimal instrumental drift over time, reflecting stable plasma conditions, consistent nebulization efficiency, and reliable ion transmission within the mass spectrometer. The close tracking of the internal standards also shows effective compensation for matrix effects and any associated signal suppression or enhancement.

Overall, these findings confirm that the analytical system maintained excellent stability over prolonged operation. The robustness of the internal standard correction method ensured that the analyte signals were accurately normalized, thereby supporting the reliability and reproducibility of quantitative results obtained during extended analytical runs.

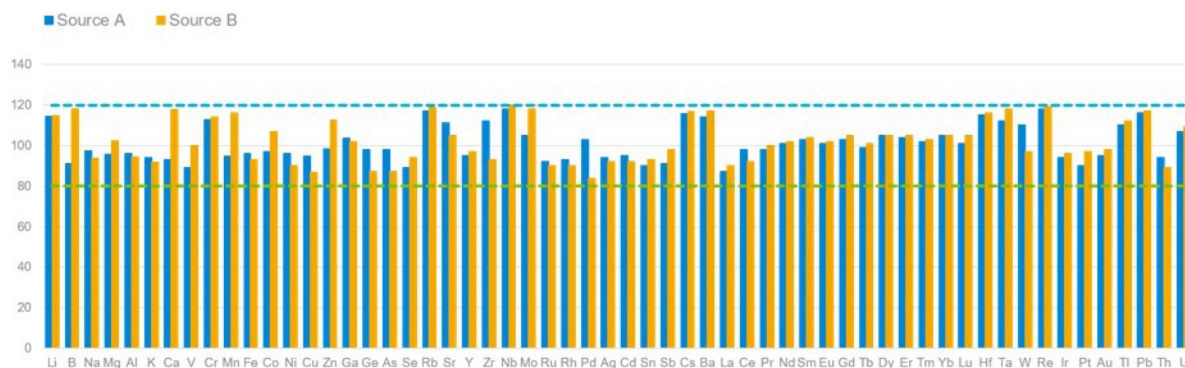


Figure 3. Spike-recovery data for 58 elements spiked at 0.1 µg/L in the two silicon sample solutions (Source A and Source B). The dotted lines indicate the 80 and 120% acceptance limits.

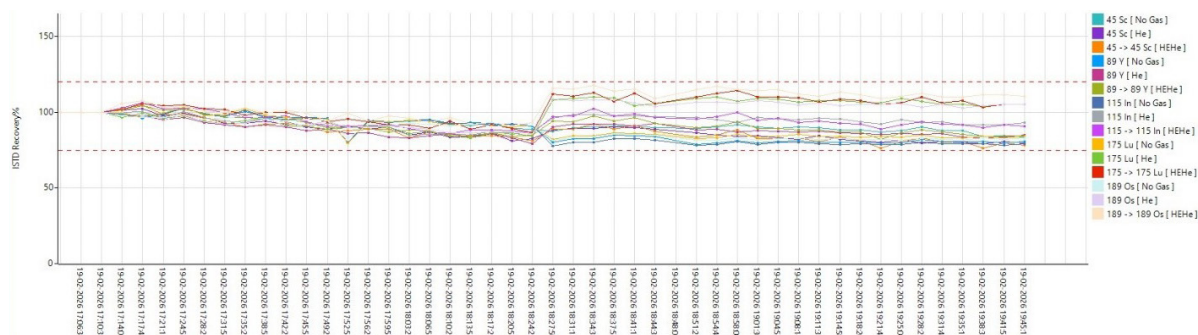


Figure 4. Internal standard stability study over three hours.

Precision and reproducibility

Precision and reproducibility of the analytical method were thoroughly assessed by performing six replicate measurements for each sample under identical operating conditions. The results for selected elements quantified in the Source B polysilicon sample are shown in Table 3.

The relative standard deviations (RSDs) were consistently less than 10%, indicating excellent repeatability and confirming stable plasma conditions, reliable ion transmission, and effective correction of matrix-related effects. The low RSD values also reflect negligible instrumental signal drift over the course of the analytical sequence, highlighting the robustness of both the instrument and the optimized method.

Table 3. Intra-day repeatability and inter-day reproducibility study of polysilicon from Source B ($n=6$ each).

Mass	Element	Repeatability (n=6)		Reproducibility (n=12)	
		Mean, µg/L	RSD, %	Mean, µg/L	RSD, %
7	Li	0.85	4.12	0.85	6.53
11	B	7.79	3.09	7.89	4.85
23	Na	19.62	1.59	19.25	2.43
24	Mg	8.52	1.17	8.35	2.33
27	Al	34.98	1.16	34.43	2.32
39	K	7.05	2.31	6.84	3.72
40	Ca	145.30	2.06	142.61	2.54
51	V	0.83	0.81	0.82	2.05
52	Cr	6.01	0.97	5.87	1.17
55	Mn	3.86	0.90	3.82	3.45
56	Fe	32.72	1.06	32.02	1.80
59	Co	1.85	1.21	1.82	1.56
60	Ni	3.98	0.99	3.99	1.82
63	Cu	1.33	1.53	1.19	1.86
66	Zn	8.69	1.09	8.44	1.74
71	Ga	0.91	1.69	0.90	1.41
72	Ge	0.76	2.53	0.76	1.68
75	As	0.66	2.32	0.68	2.14
78	Se	0.47	7.37	0.45	6.21
85	Rb	0.86	1.03	0.85	1.25
88	Sr	1.97	0.82	1.95	1.62
89	Y	0.70	4.48	0.67	5.65
90	Zr	0.92	0.81	0.92	1.30
93	Nb	0.86	1.21	0.86	1.53
95	Mo	0.94	0.97	0.94	2.26
101	Ru	0.85	1.10	0.84	1.25
103	Rh	0.79	0.84	0.79	1.32
105	Pd	0.60	5.16	0.57	2.89
107	Ag	0.98	1.11	0.98	1.77

Mass	Element	Repeatability (n=6)		Reproducibility (n=12)	
		Mean, µg/L	RSD, %	Mean, µg/L	RSD, %
111	Cd	0.50	3.08	0.49	2.63
121	Sn	0.92	0.45	0.92	0.92
125	Sb	0.80	0.88	0.80	0.92
133	Cs	0.91	0.60	0.92	0.75
137	Ba	5.68	0.89	5.15	3.04
139	La	0.74	2.73	0.73	2.55
140	Ce	0.78	2.87	0.77	2.91
141	Pr	0.83	2.02	0.81	2.92
146	Nd	0.87	1.68	0.85	3.22
147	Sm	0.88	2.12	0.86	2.92
153	Eu	0.89	1.76	0.88	2.50
157	Gd	0.97	1.52	0.95	3.03
159	Tb	0.92	1.64	0.91	2.48
163	Dy	0.92	2.06	0.91	2.96
166	Er	0.93	1.39	0.92	2.21
169	Tm	0.96	1.40	0.95	2.43
172	Yb	0.94	1.44	0.93	2.77
175	Lu	0.95	1.12	0.94	2.24
178	Hf	1.09	1.04	1.10	0.65
181	Ta	0.98	1.12	0.99	1.34
182	W	1.62	1.19	1.64	2.93
185	Re	0.99	1.46	1.00	0.99
193	Ir	0.92	1.27	0.93	1.94
195	Pt	0.87	1.39	0.88	1.60
197	Au	0.48	8.39	0.49	7.47
205	Tl	0.97	1.16	0.98	1.37
208	Pb	1.42	1.25	1.42	1.99
232	Th	0.91	2.32	0.92	2.28
238	U	1.18	1.85	1.17	2.00

Conclusion

An Agilent 8900 ICP-QQQ, configured with an s-lens ion optics system to enhance ion transmission efficiency and sensitivity, was used for the simultaneous quantification of 58 trace and ultratrace elements in two polycrystalline silicon ingot samples. Sample preparation was carried out by acid extraction in accordance with the GB/T 24582-2023 standard.

Ultratrace detection at sub-ppt levels was achieved using MS/MS (triple quadrupole) mode, utilizing multiple reaction gases to systematically mitigate spectral interferences via mass-shift and on-mass reaction pathways. This approach enabled selective removal of polyatomic interferences commonly associated with silicon matrices, thereby improving analyte selectivity and lowering background noise. The method exhibited strong analytical precision, with relative standard deviations (RSDs) below 10% across six replicate measurements, indicating consistent signal stability, efficient interference control, and robust instrumental performance.

The 0.1 µg/L spike-recovery results within ± 20% confirmed the accuracy of the quantification. The internal standard recoveries verified stable instrument performance and effective correction for matrix-induced signal variation, further supporting reliable multi-element analysis of high-purity silicon. This level of reliability is essential for maintaining accuracy at sub-ppb levels in challenging silicon matrices.

The 8900 ICP-QQQ method is well aligned with the demands of advanced manufacturing environments. It is particularly suited to PV production, where precise and reproducible impurity monitoring is vital for ensuring material quality, optimizing process control, and preserving device performance.

References

1. Kabeyi, M. J. B., & Olanrewaju, O. A. Sustainable energy transition for renewable and low carbon grid electricity generation and supply. *Frontiers in Energy Research*, **2022**, 9, Article 743114, <https://doi.org/10.3389/fenrg.2021.743114>
2. Doumon, N. Y. Transitioning to renewable energy: Challenges and opportunities. *Institute of Energy and the Environment*, **2024**, <https://iee.psu.edu/news/blog/transitioning-renewable-energy-challenges-and-opportunities>
3. Maka, A. O. M., & Alabid, J. M. Solar energy technology and its roles in sustainable development. *Clean Energy*, **2022**, 6(3), 476–483, <https://doi.org/10.1093/ce/zkac023>
4. Panagoda, L. P. S. S., Sandeepa, R. A. H. T., Perera, W. A. V. T., Sandunika, D. M. I., Siriwardhana, S. M. G. T., Alwis, M. K. S. D., & Dilka, S. H. S. Advancements in photovoltaic (PV) technology for solar energy generation. *J. Research in Technology and Engineering*, **2023**, 4(3), 30–72
5. Di Sabatino, M., Hendawi, R., & Sanchez Garcia, A. Silicon solar cells: Trends, manufacturing challenges, and AI perspectives. *Crystals*, **2024**, 14(2), 167, <https://doi.org/10.3390/cryst14020167>
6. Yang, D. (Ed.). Handbook of photovoltaic silicon. Springer, 2017, <https://link.springer.com/referencework/10.1007/978-3-662-52735-1>
7. Applications of ICP-MS: Measuring Inorganic Impurities in Semiconductor Manufacturing, Agilent publication, [5991-9495EN](#)
8. Ying, Y. Analysis of Ultratrace Impurities in High Silicon Matrix Samples by ICP-QQQ, Agilent publication, [5994-2890EN](#)
9. McCurdy, E., Woods, G., Sugiyama, N. Method Development with ICP-MS/MS: Tools and Techniques to Ensure Accurate Results in Reaction Mode, *Spectroscopy*, **2019** (9):20–27

Products used in this application

Agilent products

Product Type	Description	Part Number
Sample Introduction	Inert Kit	G4912A #001
Lens Assembly and Interface	s-lens	G3666-67400
	Platinum-tipped, nickel-based skimmer cone designed for the s-lens	G3666-67411
Chemical Standards	Multi-element calibration standard 1	8500-6944
	Multi-element calibration standard 2A	8500-6940
	Multi-element calibration standard 3	8500-6948
	Multi-element calibration standard 4A	8500-6942

www.agilent.com/chem/8900icpqqq

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